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Enhanced Heteroepitaxy

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY

LEXINGTON, MASSACHUSETTS

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FOR THE COMMANDER

Raymond L. Loiselle

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Chief, ESD Lincoln Laboratory Project Office

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MASSACHUSETTS INSTITUTE OF TECHNOLOGY
LINCOLN LABORATORY

ENHANCED HETEROEPITAXY

SEMIANNUAL TECHNICAL SUMMARY REPORT
TO THE
DEFENSE ADVANCED RESEARCH PROJECTS AGENCY

1 JANUARY – 30 JUNE 1977

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ABSTRACT

In order to determine the feasibility of using submicrometer-dimension surface-relief structures to control the orientation of deposited thin films, a technology for fabricating the required structures is being developed. The technology involves holographic lithography, x-ray lithography, and reactive ion etching. A recently developed process for fabricating polyimide-membrane x-ray-lithography masks with thicknesses ranging from 0.5 to several micrometers, and measurements of distortion in such masks are reported. Techniques for producing high-aspect-ratio, vertical-walled relief gratings of 1600-Å linewidth with smooth line edges in SiO_2 on Si substrates are described. Soft x-ray lithography (13.3 to 44.7 Å) is first used to expose relief structures in polymethyl methacrylate (PMMA). Liftoff of chromium and reactive ion etching in CHF_3 gas are then used to transfer the structure into the SiO_2. The line-edge smoothness and corner sharpness of surface-relief structures fabricated using x-ray lithography and reactive ion etching were evaluated using the distribution of deposited gold islands. Line-edge smoothness of the order of 100 Å was achieved. Corner sharpness was estimated to be less than 50 Å.

CONTENTS

INTRODUCTION

The objectives of this research program during calendar year 1977 are to:

(1) Determine the feasibility of using submicrometer-dimension surface-relief structures to control the orientation of a variety of deposited thin films,

(2) Determine if single-crystal films of low defect density can be produced using artificial surface-relief structures, and

(3) Determine if device quality AlN or ZnO films can be produced on SiO_2 or Si_3N_4 over Si.

The tasks within this program include: (1) the development of a technology for fabricating the required submicrometer surface-relief structures, (2) the deposition of thin film material, and (3) the analysis of structures fabricated and their influence on thin film growth and orientation.

This report consists of three sections that describe progress in the first half of calendar year 1977. Sections I and II cover progress on the development of surface-relief structures under task (1). Section III describes a study of the early stages of gold growth on surface-relief structures in SiO_2, which was carried out as part of task (2).

I. POLYIMIDE-MEMBRANE X-RAY-LITHOGRAPHY MASK

X-ray-lithography masks consist of an absorber pattern on a thin membrane that is relatively transparent (i.e., $\lesssim$3-dB attenuation) to soft x-rays. Several materials have been used as transmitter membranes including silicon,[1,2] Al_2O_3 (Refs. 3 and 4), SiO_2/Si_3N_4 (Refs. 5 and 6), mylar,[7] and polyimide.[8,9]

We describe here a recently developed process for fabricating polyimide-membrane x-ray-lithography masks, with thicknesses ranging from 0.5 to several micrometers, and measurements of distortion in such masks. The process produces large-area rugged x-ray masks suitable for use at the 13.3-Å Cu_L wavelength, as well as at the 44.7-Å C_K and 8.34-Å Al_K wavelengths. The polyimide surface is optically smooth, and the membrane is transparent to visible radiation.

In the first step of the process, a glass substrate is coated with a film of polyamic acid (Dupont product PI-2530) using a conventional spinning technique. Thicknesses of 0.5 to 5 μm are readily obtained by varying the spin speed and/or the dilution of the polyamic acid. For a film thickness of 1 μm, a dilution of 4 parts PI-2530 in 1 part N-methyl-2-pyrollidone and 1 part acetone, and a spin speed of 6000 rpm are used. The polyamic acid is converted to polyimide by curing at 150°C for 15 min. and then at 250°C for 60 min. The desired x-ray-absorber pattern is then formed in gold on top of the polyimide by using any lithographic means such as photolithography, electron-beam, or holographic lithography. An important feature of the process is that the polyimide membrane remains attached to a glass substrate during fabrication of the absorber pattern. The glass serves as a heat sink and thus avoids the problem of heating during ion-beam etching or other processes. The polyimide is relatively inert and stands up to most chemical-etching environments.

A support ring and a copper tube are bonded to the polyimide using epoxy as indicated in Fig. 1(a). The glass substrate is then etched by immersing the assembly in a solution of hydrofluoric acid as shown in Fig.1(b). During etching, the copper tube is filled with isopropyl alcohol to provide a positive pressure against the polyimide and to prevent damage from entry of acid through pinholes or diffusion through the membrane. After etching of the glass substrate, the support ring is separated from the copper tube by cutting the polyimide between the two. Figure 2 is a cross-sectional schematic of a completed x-ray mask. The aluminum contact is evaporated onto the mask after the glass etching. It acts as an infrared reflector and as an electrical contact in an electrostatic mask-contacting scheme.

Using the holographic moiré method illustrated in Fig. 3, we have measured the inhomogeneous distortion of 9000-Å-thick polyimide-membrane masks on which a 3200-Å spatial-period gold grating had been fabricated using holographic lithography and ion-beam etching. The gold thickness was 1000 Å and between it and the polyimide there was a 100-Å-thick chromium film. When the mask is placed back in the holographic configuration, as illustrated in Fig. 3, any inhomogeneous distortion perpendicular to the grating lines will alter the direction of the diffracted beams (indicated as dotted lines in the figure) and lead to interference between them and the beams transmitted through the mask (indicated as solid lines in the figure). If a screen is placed in the region where the two beams interfere, the mask distortion will be displayed as a two-dimensional fringe pattern.

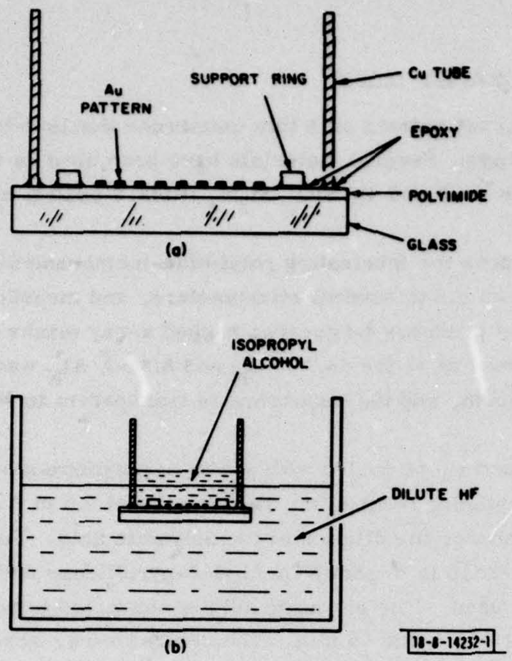

Fig. 1. Fabrication of a polyimide-membrane x-ray-lithography mask: (a) cross-sectional illustration showing support ring and copper tube bonded to polyimide membrane and (b) method used to protect absorber pattern during etching of glass substrate under polyimide membrane.

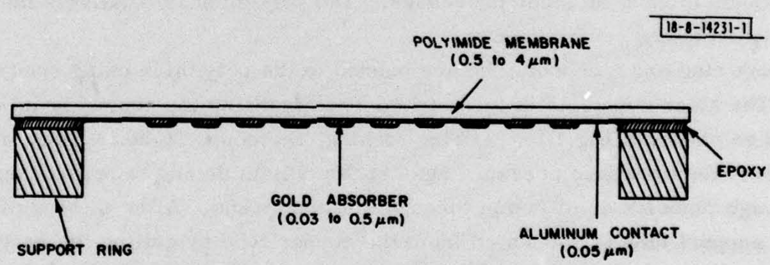

Fig. 2. Cross-sectional illustration of polyimide-membrane x-ray-lithography mask.

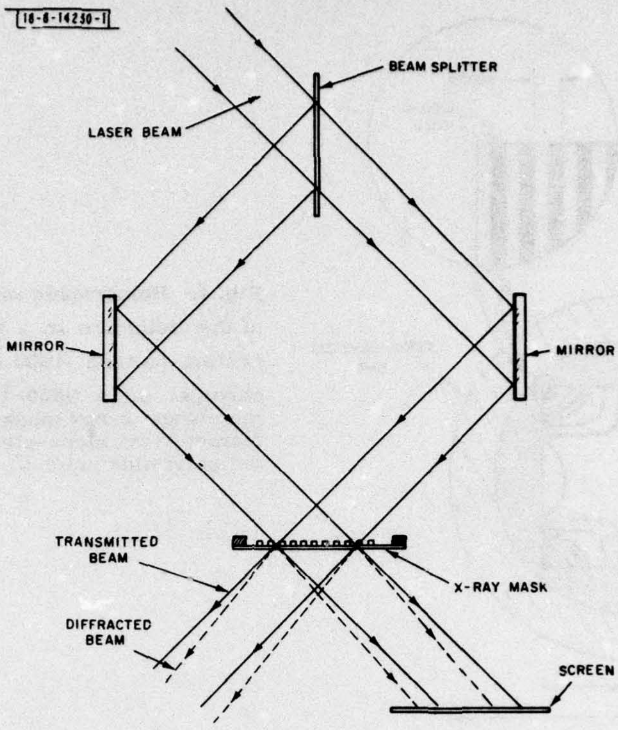

Fig. 3. Holographic moiré method for displaying distortion in a grating pattern on an x-ray mask.

Two methods for mounting the polyimide membrane to the ring were used. In one, the ring was bonded to the polyimide prior to glass etching; in the other, the bonding was done after the glass etching. Our distortion measurements were made immediately after mask preparation. We did not attempt to study long-term stability or possible changes induced by exposure to x-rays. Figure 4 shows a typical fringe pattern display of the distortion, for the case of ring bonding prior to glass etching. The important feature is the large area (about 7.5 mm in diameter) where no fringes occur, indicating that the distortion is less than 1600 Å (half the grating period). This corresponds to a fractional distortion less than 2 parts in 10^5. For comparison, the best performance for a scanning electron-beam-lithography (SEBL) system is 1000-Å distortion over a 2- × 2-mm field, or a fractional distortion of 5 parts in 10^5. Approximately the same membrane distortion was observed if the stainless-steel ring was bonded to the polyimide after etching of the glass, provided that care was taken not to stretch the polyimide inhomogeneously. If the polyimide was somewhat carelessly stretched during ring bonding (early stages of technique development), an inhomogeneous distortion of 1 part in 10^3 was measured by the method depicted in Fig. 5.

The fringe pattern in Fig. 4 is interpreted as reflecting the stress pattern of the square area of gold grating lines. It is well-known that metal films are typically under tensile stress. However, this stress can sometimes be made zero by adjusting the parameters of deposition or by

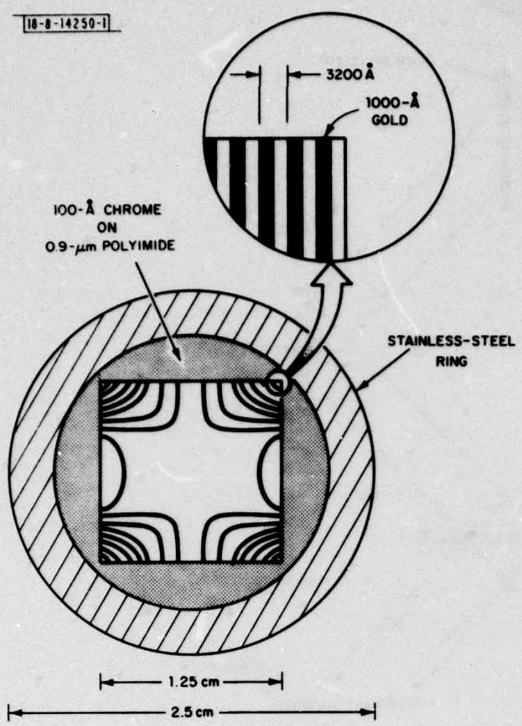

Fig. 4. Holographic moiré fringe display of the distortion in a 3200-Å period gold grating pattern (1000 Å thick on 100-Å chrome) on a 9000-Å-thick polyimide-membrane x-ray mask when the 2.54-cm-diameter stainless-steel ring is bonded to the polyimide prior to glass etching.

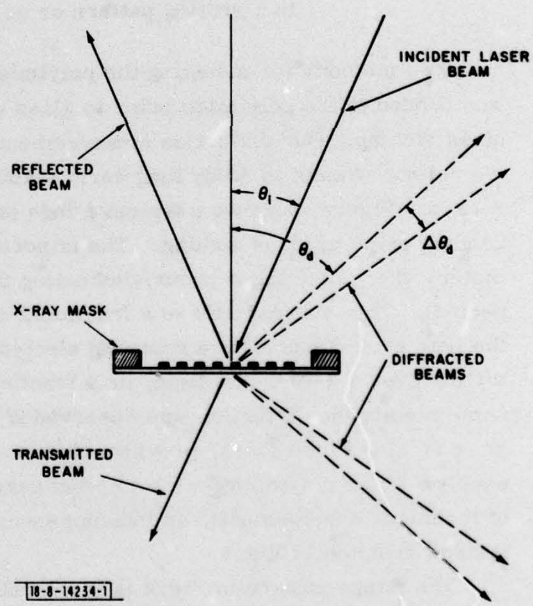

Fig. 5. Schematic illustration of method of measuring distortion of a grating pattern from the angle of diffraction. Homogeneous distortion is equivalent to a change in the average grating period from that originally generated, and is readily measured from the angle of diffraction. Inhomogeneous distortion leads to a spread, $\Delta\Theta_d$, in the angle of diffraction.

using appropriate multilayers. We have not attempted to do this but feel it would be a straightforward engineering task to develop methods for depositing films that were effectively stress-free.

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II. SURFACE-RELIEF STRUCTURES WITH LINEWIDTHS BELOW 2000 Å

The influence of a submicrometer surface-relief structure on the orientation of an overlayer film is expected to increase as its spatial period is reduced. In many cases, well-defined vertical-sidewall square-wave-cross-sectional profiles are required. The fabrication of such structures by the planar process requires lithographic methods for the controlled exposure of relief structures in resist as well as means for etching relief structures into substrate surfaces. In this section, we report on the use of Cu_L and C_K soft x-ray lithography to expose grating relief structures having 1600-Å linewidths and sharp vertical sidewalls in PMMA resist films on thick substrates as well as on the use of rf reactive ion etching to achieve vertical-walled relief structures in thermal SiO_2 on silicon.[10]

For exposing linewidths below 2000 Å in resist films, x-ray lithography has a number of fundamental and practical advantages over SEBL and holographic lithography. On membranes of the order of 100 Å thick, linewidths below 100 Å have been written by SEBL using in situ polymerization of mobile monomers adsorbed on the membrane surface[11,12] (so-called contamination writing). However, this resolution cannot be achieved on thick substrates due to electron backscattering, and writing times are extremely long for the contamination writing process. SEBL also can be used to expose an electron resist film on a substrate but, again, electron backscattering leads to a high background level, and this severely limits the exposure latitude for gratings having spatial periods below about 4000 Å (Ref. 13). Moreover, because the electron energy dissipation in the resist as a function of lateral distance varies with depth in the resist, one does not have freedom to control the cross-sectional structure developed in a resist film. In particular, it is difficult and perhaps impossible to produce vertical sidewalls in grating structures having spatial periods below about 4000 Å. Other difficulties with SEBL are limited number of pattern lines (about 4000 to 16,000 lines per pattern area), pattern distortion, electrical charging, and possibly radiation damage.[14]

Holographic lithography is a simple low-distortion large-area pattern generation technique suitable for periodic and quasi-periodic patterns. Spatial periods as fine as 1100 and 835 Å have been produced using a He-Cd and an upconverted Nd:YAG laser, respectively.[15,16] However, vertical sidewalls cannot be produced in resist films because of standing waves produced by back reflection from the substrate. Some control over sidewall profiles can be exercised by manipulating the nonlinearity of the resist recording film but, in general, rounded profiles are obtained and there is usually substantial edge raggedness.

The intrinsic resolution of soft x-ray lithography is given by the effective range of the photoelectrons excited in a resist film upon absorption of an x-ray photon. This is estimated to be 200 Å for the Cu_L soft x-ray (13.3 Å) and 50 Å for the C_K x-ray (44.7 Å) (Ref. 5). The cross-sectional profile obtained in high-contrast resists such as PMMA depends on the exposure geometry (primarily the penumbra and the angle of incidence in the case of a conventional electron bombardment x-ray source) and the resist development properties. One has considerable freedom to adjust sidewall angles by varying the angle of incidence, the penumbra, and the exposure and development times.

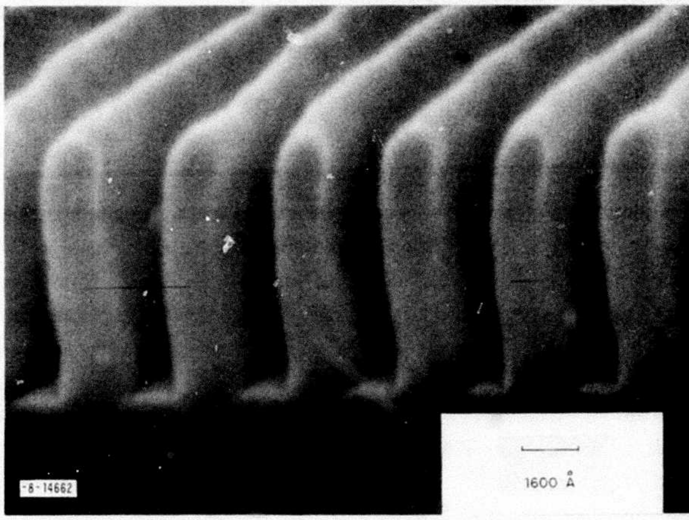

Fig. 6. Scanning electron micrograph of the cross section of a 1600-Å-linewidth grating pattern exposed in a 8500-Å-thick PMMA film on an SiO_2/Si substrate using Cu_L x-radiation at 13.3 Å. The slight curvature of the grating lines was probably due to softening of the PMMA caused by overdevelopment or heating during gold coating prior to microscopy in an SEM.

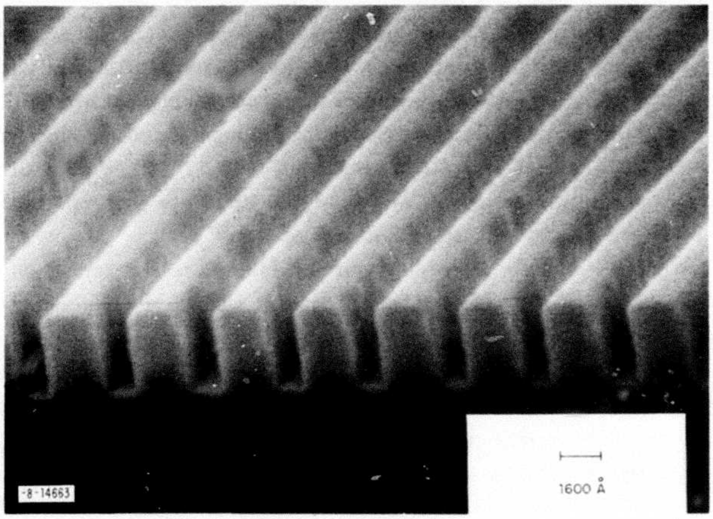

Fig. 7. Scanning electron micrograph of the cross section of a grating pattern etched into SiO_2 on an Si substrate by reactive ion etching in CHF_3 gas. The groove width is about 900 Å.

Figure 6 illustrates the sharp vertical sidewalls and large aspect ratios that can be obtained using Cu_L x-radiation and PMMA resist. Similar results were obtained using C_K x-radiation (44.7 Å), but due to the higher intrinsic resolution (~50 Å) of the C_K radiation, raggedness of the order of 100 Å along line edges in the mask was reproduced in the resist.[17] The Cu_L radiation, on the other hand, tends to smooth out such fine detail and thereby produces smooth edges. The mask used to produce the result in Fig. 6 consisted of a 3200-Å spatial-period grating pattern in gold 1000 Å thick on a 0.9-μm-thick polyimide membrane fabricated using the method described in Sec. I above. The polyimide-membrane mask was held in intimate contact with substrates by applying a DC voltage between an aluminum film on the membrane and the substrate (usually an SiO_2-coated silicon wafer). No filter is used between the x-ray source and the substrate although the aluminum film serves to reflect infrared radiation from the source.

The x-ray source was operated at 400 W and both 8 and 6.5 kV. For a 4200-Å-thick film of PMMA to be completely developed in 1 min. in a mixture of 40% methyl isobutyl ketone and 60% isopropyl alcohol, the required exposure times were 75 and 68 min., respectively, for the two operating voltages. Resist adhesion was superior if the lower voltage was used. These results indicate that for a given amount of characteristic radiation absorbed, the deleterious effects of the hard continuum radiation emitted from the copper target are less pronounced for the lower voltage. A calculation indicated that at 8 kV the total integrated power absorbed from the continuum was only 5 percent of that absorbed from the Cu_L line.

In order to etch vertical-sidewall relief gratings into the substrate surface, reactive ion etching was used. With this method, a directional or anisotropic etching is possible and the problem of redeposition of material sputtered from the substrate surface is avoided. This latter problem is characteristic of ion-beam etching (so-called ion milling) and rf-sputter etching, and limits both the dimensional control and the sidewall angles that one can achieve.[18,19] The reactive ion etching method is described in detail elsewhere.[20] In brief, we used a conventional rf-sputter etching system at a frequency of 13.56 MHz and an rf power density of 0.3 W/cm^2. Etching was done in CHF_3 gas at a pressure of 10^{-2} Torr and a flow rate of 15 cm^3/min. Figure 7 illustrates a sharp vertical-sidewall relief grating etched into SiO_2. The groove widths are approximately 900 Å.

The material PMMA, such as in the resist pattern in Fig. 6, is etched at a rate of about 280 Å/min. while SiO_2 is etched at a rate of about 200 Å/min. Thus, in order to etch vertical-sidewall grooves into the SiO_2, a more suitable mask is required. We used a chromium mask because it is etched at only about 12 Å/min., has a good adhesion, and is fine-grained. The chromium grating was produced by the liftoff process using a resist pattern similar to that shown in Fig. 6, but where the PMMA was only 4200 Å thick.

In summary, we have demonstrated that soft x-ray lithography using Cu_L x-radiation is an effective means of obtaining smooth-edged vertical-sidewall high-aspect-ratio relief gratings of 1600-Å linewidth in PMMA resist, and that such structures also can be etched into SiO_2 substrates by reactive ion etching in CHF_3 gas.

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III. STUDIES OF GOLD ISLAND GROWTH ON SUBMICROMETER SURFACE-RELIEF STRUCTURES

Many researchers have observed that when thin films are deposited on a substrate, nucleation occurs preferentially along natural cleavage steps and surface defects.[21,22] Micrographs obtained by Shimaoka and Komoriya[23] suggest that tin nuclei contacting the natural cleavage steps on NaCl are predominantly of a single orientation, while those nucleated away from the steps exhibit more or less random orientation. In most types of heteroepitaxial film growth (meaning systems such as Si on Al_2O_3 and excluding $Al_xGa_{1-x}As$ and similar systems), the nuclei that first form at separation distances of the order of several hundred angstroms have a multiplicity of crystallographic orientations. The single orientation that we call epitaxy does not occur until the islands that grow from the nuclei reach sizes of the order of 1000 Å in diameter. At that point they coalesce and induce recrystallization in one another, resulting in a continuous film of a single orientation.[21,22,24] Steps on a surface can act as a border to a growing island. Distler et al.[25] believe that heteroepitaxy is dependent on point defects that naturally form a regular matrix of intersecting lines on a surface. In most types of homoepitaxy (e.g., Si on Si, GaAs on GaAs), substrates are oriented 2 or 3 degrees off axis. The resulting natural terraces on the substrate surface are separated by distances of the order of 100 Å and have a controlling influence on the subsequent homoepitaxial growth.[26,27] The above observations and theory indicate that steps and point defects have an important effect on thin film growth, and this in turn suggests that one may be able to exert some form of control over such film growth by means of artificially created surface-relief structures such as gratings, grids or regular arrays of point defects.

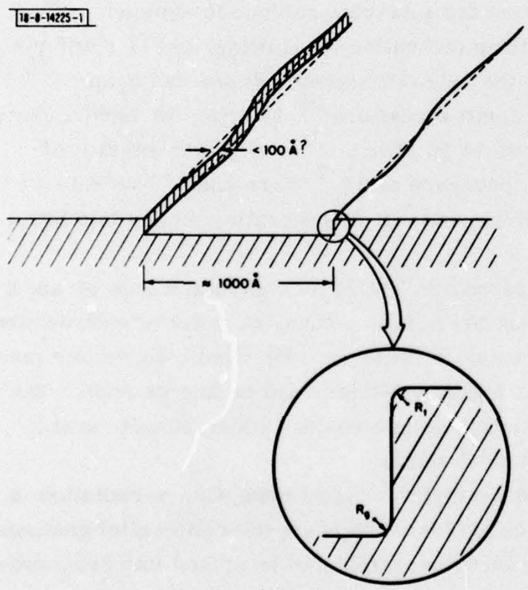

Fig. 8. Schematic illustration of an idealized surface-relief structure for the study of thin film nucleation and growth.

Figure 8 illustrates an idealized relief structure which could be used for studies of the influence of artificially created surface-relief structures on the growth and orientation of thin films. Developing the technology for producing such structures has been the major emphasis of our work to date. The important features to note about the Fig. 8 structure are the radii of curvature, R_g and R_t at the base and at the top of the steps, the vertical sidewall, the smoothness

along the length of the steps, and the periodicity of the grating. In order to influence strongly nucleation and growth, it appears that R_g should approach 10 Å, the sidewalls should be as nearly vertical as possible, the edge smoothness should approach about 100 Å and be free of "fast ripple," and the grating period should be less than about 4000 Å.

Our approach to fabricating submicrometer surface-relief structures approaching such specifications is the planar process, which begins with the exposure of a relief structure in a resist film and then uses this either as an etching mask or as a mask for a liftoff process. The resist exposure method must be capable of exposing, on semi-infinite substrates, gratings with spatial periods below 4000 Å, while controlling the sidewall angle. For this, x-ray lithography is preferred over electron-beam lithography or holographic lithography as discussed in Sec. III.

Using resist structures exposed by Cu_L x-ray lithography, we have produced relief gratings on SiO_2 and Si_3N_4 films over silicon wafers by liftoff of evaporated SiO_x and carbon, by ion-beam etching, and by reactive ion etching. The SiO_x and carbon gratings were only about 100 Å thick. We believe that their cross-sectional structure was sharp cornered at the base (i.e., small R_g). However, this is difficult or nearly impossible to evaluate directly by electron microscopy. Instead, we used a decoration technique in which gold was ion-beam sputtered onto the structures to a thickness such that discrete islands were formed but the film was not continuous. We then interpreted the aspects of the relief structure, such as R_g and edge smoothness, from the pattern of gold islands. To date, this study is somewhat inconclusive with respect to R_g, but we have determined that edge smoothness of the order of 100 Å has been achieved. In order to see the pattern of gold islands, the sample was thinned and viewed in transmission by TEM. Figure 9 shows the sequence of steps involved in the decoration technique.

Relief structures produced by ion-beam etching are not expected to have the idealized cross section depicted in Fig. 8. This is due primarily to the problem of redeposition.[18,19] Nevertheless, we have etched gratings by ion-beam etching to a depth of 10 Å and studied them by the decoration technique. The edge smoothness and the value of R_g appeared to be equivalent to what was obtained by liftoff.

As described in Sec. II, reactive ion etching avoids the problem of redeposition while retaining directional etching. Decoration experiments on grooves etched to a depth of 100 Å by reactive ion etching in CHF_3 yielded the results shown in Fig. 10.

Clearly, the density of gold islands is lower in the vicinity of the groove edges than elsewhere in the grating. We believe this "antidecoration" is due to a coalescence mechanism rather than a depressed nucleation rate. When two islands coalesce, their common center-of-gravity remains essentially fixed in position; however, their individual centers-of-gravity move toward one another. Thus, islands located along the groove edges are drawn toward the middle of the grooves leaving behind a void. In some cases, we detected an enhanced nucleation along the groove edges. Such enhanced nucleation is expected when the contact angle is $\lesssim 100°$ and the radius of curvature at the base, R_g, is comparable to the size of the critical nucleus. We estimate that R_g is less than 50 Å.

Our studies of the influence of surface-relief structures on the nucleation, growth, and orientation of thin films are at an early stage. As indicated above, some influence has already been demonstrated. Future work will concentrate on demonstrating an influence on orientation using materials that ordinarily exhibit (100) texture on amorphous substrates.

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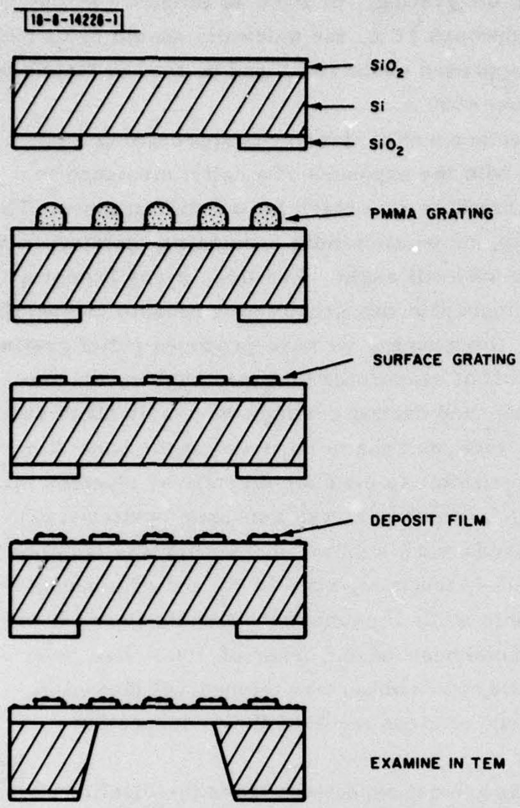

Fig. 9. Sequence of steps illustrating the creation of a relief grating structure on an SiO_2 substrate, the decoration of that structure by nuclei of a deposited film and the thinning of the sample by selective etching of the silicon so that the decorated structure can be viewed in transmission in an electron microscope.

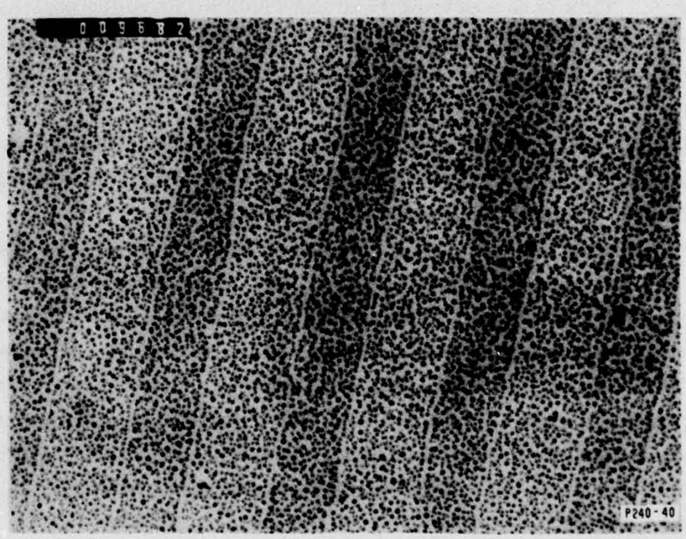

Fig. 10. Conventional electron micrograph of gold island decoration of a grating relief structure etched 100 Å into an SiO_2 substrate using reactive ion etching. Note that the pattern of islands clearly delineates the grating lines. The grating spatial period is 3200 Å, or approximately 1600-Å linewidth.

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In order to determine the feasibility of using submicrometer-dimension surface-relief structures to control the orientation of deposited thin films, a technology for fabricating the required structures is being developed. The technology involves holographic lithography, x-ray lithography, and reactive ion etching. A recently developed process for fabricating polyimide-membrane x-ray-lithography masks with thicknesses ranging from 0.5 to several micrometers, and measurements of distortion in such masks are reported. Techniques for producing high-aspect-ratio, vertical-walled relief gratings of 1600-Å linewidth with smooth line edges in SiO_2 on Si substrates are described. Soft x-ray lithography (13.3 to 44.7 Å) is first used to expose relief structures in polymethyl methacrylate (PMMA). Liftoff of chromium and reactive ion etching in CHF_3 gas are then used to transfer the structure into the SiO_2. The line-edge smoothness and corner sharpness of surface-relief structures fabricated using x-ray lithography and reactive ion etching were evaluated using the distribution of deposited gold islands. Line-edge smoothness of the order of 100 Å was achieved. Corner sharpness was estimated to be less than 50 Å.

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